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H. Marshak and R. B. Dove

Heat Division
Institute for Basic Standards
National Bureau of Standards
Washington, D.C. 20234

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A Right Angle ^{3}He Cryostat Incorporating a High Field Superconducting Solenoid

H. Marshak and R. B. Dove

Construction and operation of a novel ^{3}He cryostat, incorporating a large superconducting solenoid mounted at right angles to the cryostat's vertical axis, is described. This new cryostat which is part of the transportable National Bureau of Standards ^{3}He refrigerator, has been used successfully for nuclear orientation studies at the Atomic Energy Research Establishment, Harwell, England.

Key words: ^{3}He cryostat; ^{3}He refrigerator; nuclear orientation; superconducting solenoid.

1. INTRODUCTION

In designing a ^{3}He refrigerator,[1/] for nuclear orientation studies a problem often encountered is incorporating a high field superconducting solenoid when nuclear polarization is required. The usual solution for thin samples (< 0.5 in.) is to use a split solenoid where one then has vertical access for the cryostat tails (which contain the sample) and horizontal access (with a minimum of material) for the beam. In the case where one needs a thick polarized sample, for example, in some types of neutron experiments, the sample is of the order of 5 in. thick, the bore of the superconducting split solenoid has to be very large and the field is reduced considerably. Although the field can be increased by adding more turns, this becomes very difficult and expensive when high

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1/ The terminology which we will use in this paper is to call the entire system (cryostat, pumps, vacuum gauges, etc.) a refrigerator, whereas the part which contains the cryogenic liquids and where the actual cooling takes place is called the cryostat.

fields (>~4.0 MA/m)[2] are required. A solution to this problem is to use a straight superconducting solenoid mounted horizontally. This approach, although simplifying the magnet requirements[3] as well as the sample mounting and window geometry, does add to the complexity of construction as the ^{3}He cryostat must then have right angle tails.

The purpose of this paper is to describe the construction and operation of such a ^{3}He cryostat. In addition to incorporating a high field superconducting solenoid mounted horizontally, the following design criteria were imposed:

1. The new ^{3}He cryostat should be compatible with the existing ^{3}He refrigerator [1]* which is of the recirculating type. This refrigerator was designed to be easily transportable and self-contained as well as cope with a large heat flow.
2. It should have large enough cryogenic baths so that it can be operated continuously at temperatures as low as 0.3 K for periods of 24 - 48 hours with a minimum of attention. This condition is dictated by the fact that most particle accelerators are operated 24 hours a day and it is difficult and "expensive" to get into the target area once the machine is running.
3. Finally, the design should be such that the "working end" (^{3}He cooled region) of the cryostat can be easily modified for other types of experiments in this temperature region.

The ^{3}He cryostat described below was initially designed to accommodate a thick (~ 5 in.) holmium-165 sample and a relatively high field (4.38 MA/m) superconducting solenoid.

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* Figures in brackets indicate the literature references at the end of this paper.

2/ 1 MA/m = 12.57 kOe.

3/ Large bore-high field (>~ 8.0 MA/m) superconducting solenoids are readily available.

2. DESCRIPTION OF APPARATUS

A schematic drawing of the cryostat is shown in figure 1. The overall length is 79 in. and its outer diameter is 11.5 in. It is constructed mainly from Type-347 stainless steel except for those parts where high conductivity is needed; these are constructed from HPOF (high-purity oxygen-free) copper. The construction philosophy was to helium-arc weld whenever possible, if this was not possible then hard-solder was used, and finally if this was not possible soft-solder was used. For the few low temperature joints which have to be opened frequently we used a low melting soft-solder rather than soft-metal O-rings. Various components of the cryostat were helium leak detected as they were constructed, and whenever possible, at their operating temperature (e.g. 77 K, 4.2 K, etc.). Thus, the time required for leak testing and "debugging" the assembled cryostat was reduced considerably.[4/]

The cryostat has two separate liquid nitrogen baths, a large one having a capacity of 16 liters and a small one with a capacity of 3.5 liters. Whereas the large bath and its extended copper radiation shield serve the usual purpose, the small bath serves as both a radiation trap and a thermal ground at 77 K for all the tubes going into and making up the main ^{4}He bath. The latter, when normally filled to a distance of 4 in. from the bottom of the small nitrogen bath, contains 18 liters. A small ^{4}He bath having a capacity of 1.2 liters houses the superconducting solenoid. This bath is connected to the main 4.2 K bath by three 0.5 in. i.d. copper "solenoid bath feed tubes." The entire 4.2 K liquid helium system is closed so that the gas, which normally passes into the atmosphere through a blow off valve, can be recovered if necessary. The pumped ^{4}He bath has a capacity of 1.6 liters and serves both to liquefy the returning ^{3}He gas and as a 0.9 K radiation shield surrounding the ^{3}He tail and holmium sample. Gas from the exhaust side of the ^{4}He pumps passes through a gas meter so that the evaporation

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4/ The ^{3}He refrigerator operated successfully the first time it was tested.

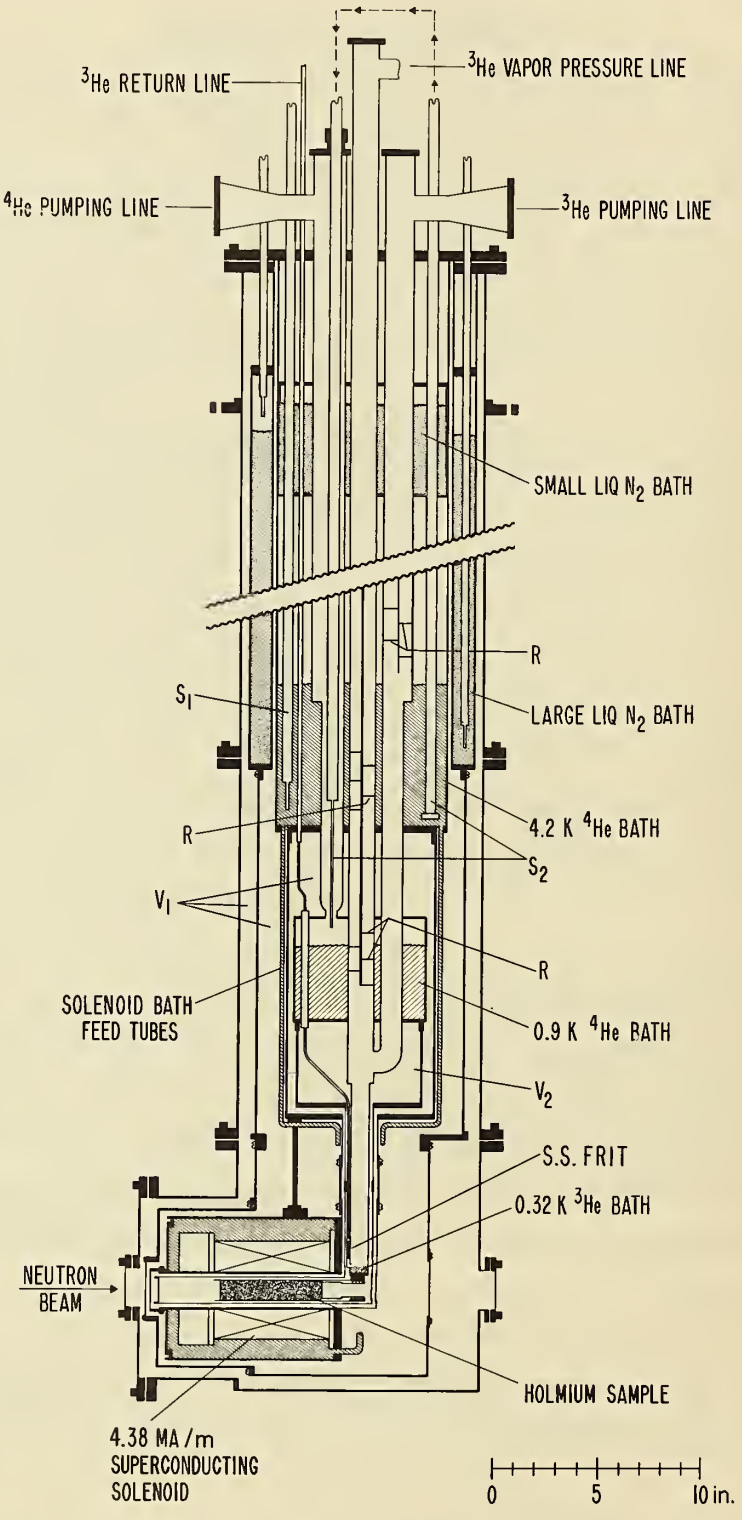

Fig. 1. Schematic view of the ^{3}He cryostat. V_1, main vacuum space; V_2, sample vacuum space; S_1, main ^{4}He bath transfer tube; S_2, pumped ^{4}He bath transfer tube; R, radiation shields.

rate can be monitored continuously. This gas can also be recovered if necessary. The ^{3}He system which is, of course, closed, contains 3 liters at NTP. Under ordinary operating conditions, that is when the sample is at 0.32 K and the ^{3}He gas is being continuously circulated, there is approximately 3 cc of liquid in the ^{3}He bath. The ^{4}He and ^{3}He pumping tubes, the ^{3}He return tube, and the ^{3}He vapor pressure tube are labelled at the top of the cryostat in figure 1. The latter tube is used to determine the temperature of the ^{3}He liquid. In the ^{3}He return tube there is a condenser packed with a very fine copper wool located in the 0.9 K bath. The sintered stainless steel frit is used to restrict the flow of ^{3}He liquid. Some of the radiation shields, R, in these main tubes are also shown. The transfer syphon S_1 for the main ^{4}He bath extends (although not shown in this figure) to the very bottom of the superconducting solenoid bath by means of a 0.125 in. i.d. teflon tube. This extension serves a dual purpose — first, to remove all of the liquid nitrogen which is used in precooling; and second, to yield a greater transfer efficiency in filling the magnet and main ^{4}He bath with liquid helium. The small syphon S_2 is U-shaped and connects the pumped ^{4}He bath with the main ^{4}He bath. There is a valve composed of a 0.125 in. diameter sapphire sphere with a gold seat, located at the end of S_2 in the 4.2 K bath. This small syphon enables us to transfer liquid helium during the main transfer without using a second storage dewar, as well as retransferring (if necessary) from the main bath at a later time.

The cryostat's main vacuum space V_1 is common to the liquid nitrogen baths and the 4.2 and 0.9 K liquid helium baths (there are baffled holes in the radiation shields). There is a layer of activated charcoal over most of the top of the 0.9 K bath which acts as a cryopump for V_1 once this bath is cold. A second independent vacuum space V_2 is common only to the bottom of the 0.9 K bath, the ^{3}He tail and holmium sample. ^{3}He exchange gas is used in it to cool the ^{3}He tail and holmium sample to $\sim$ 1 K. This exchange gas is, of course, pumped away before the ^{3}He system is started. The valves and common pumping system

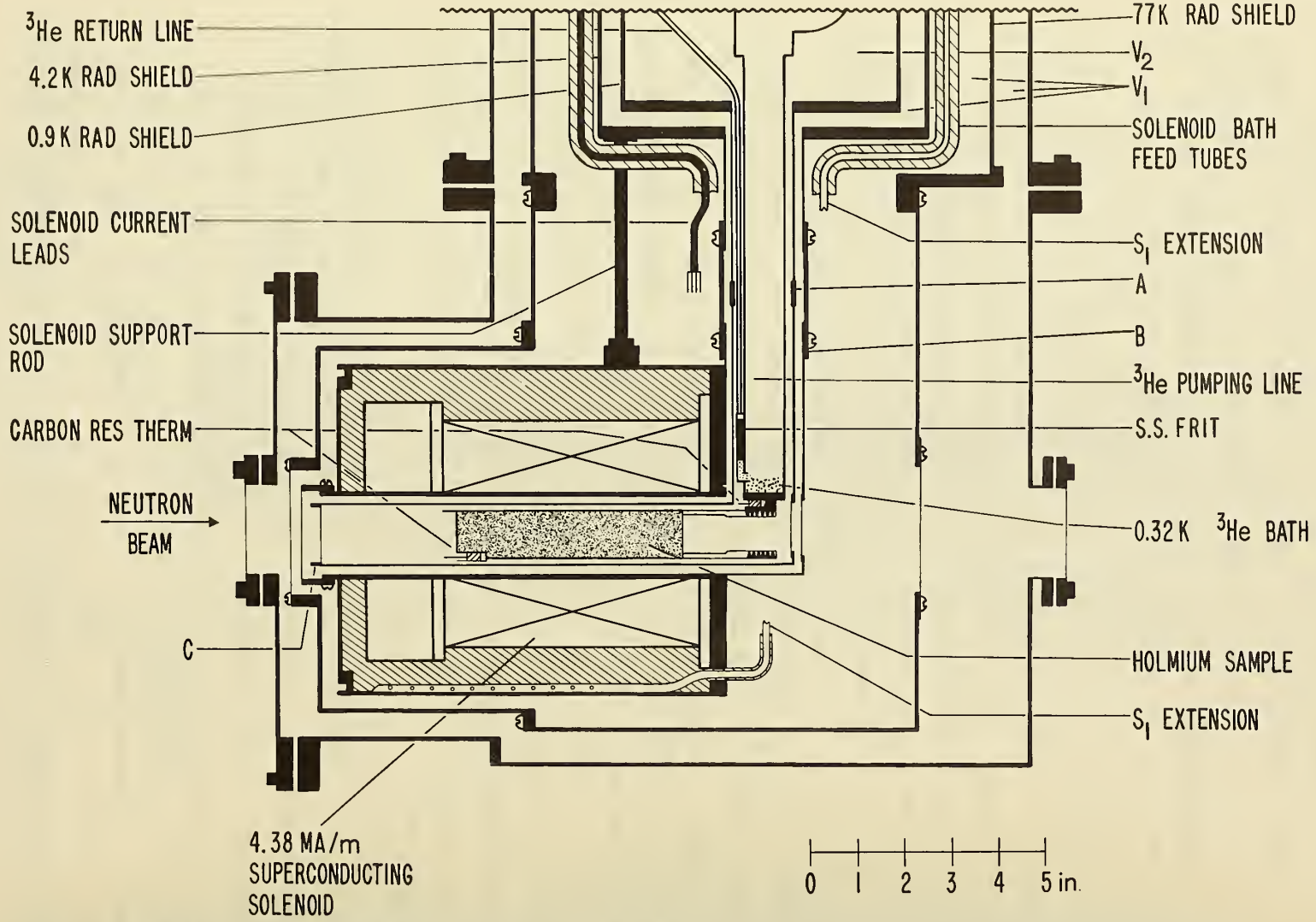

Fig. 2. Detail view of the lower part of the ^{3}He cryostat showing the right angle geometry. V_1, main vacuum space; V_2, sample vacuum space; S_1, main ^{4}He bath transfer tube; A, solder joint (vacuum); B, sleeve joint (mechanical); C, window solder joint vacuum.

for V_1 and V_2 are not shown in figure 1. During actual operation of the refrigerator the valves to V_1 and V_2 are closed. The pumping systems for the ^{3}He and ^{4}He baths have been described previously [2] and will not be described here.

Since the novel feature of the cryostat is the lower part containing the horizontally-mounted superconducting solenoid, it is worthwhile to describe the construction and assembling procedure for this part in greater detail, see figure 2. The bottom 3.25 in. of the ^{3}He tail is machined from one piece of copper and terminates in a threaded hole (0.875 in. - 16) to accept the sample holder. There are two resistors mounted in the ^{3}He tail above the threaded hole. One of these is a 470 Ω Speer carbon resistor (grade 1002) which is used as a secondary thermometer. The other one (not shown in the figure) is a 10 kΩ - 1 W metal film resistor which is used as a heater. The bottom surface of the inside of the ^{3}He tail has a thin copper ribbon (wound in the form of a spiral) soldered to it in order to increase the contact area of the ^{3}He liquid — thereby reducing the "Kapitza resistance." The holmium sample in the form of a cylinder (4.7 in. long and 0.875 in. in diameter) is soldered [5] into the tubular copper sample holder. This is threaded into the ^{3}He tail using Apeizon-N grease to improve the thermal contact.[6] A second 470 Ω Speer resistor is mounted in the front face of the sample (out of the neutron beam) and is used to monitor the sample's temperature.

In order to get the horizontally mounted superconducting solenoid as close to the ^{3}He tail as possible, we used zero radius of curvature elbows (square elbows). For this reason the 0.9 K right-angle radiation shield (from A on down) and the solenoid bath (from B on down),

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[5] Using Indalloy #2 solder (Indium Corporation of America) without any flux.

[6] Apiezon-N grease was used on all the mechanical joints of the radiation shields to improve the thermal contact.

although at different temperatures and thus cannot be in contact when operational, are not independent and represent a single unit in assembling. In addition the 0.9 K radiation shield must be leak tight as it encloses the vacuum space V_2. It is constructed from 1.250 in. o.d. by 0.0625 in. wall copper tubing which has been cut at 45° and then welded to form a square elbow. However, before it is welded, the machining of the solder cup joint at A, the machining at C to accept the thin soldered window, and the "thinning" of the tube to 0.020 in. for the rear window have all been done.[7] After it is welded, two 1.625 in. o.d. by 0.0625 in. wall, stainless steel tubes — also cut at 45° — are slipped over it and welded, again, all machining of the stainless steel tubes having been done prior to the welding. From this point on in the construction, the 0.9 K radiation shield cannot be removed from the solenoid bath housing. Next the back plate of the solenoid bath is slipped on and welded on the inside. The outer solenoid bath tube is then welded on. At this point the superconducting solenoid is secured to the back plate. With the three feed tubes on the solenoid bath temporarily sealed off, it is immersed in water with the face plate exposed, the latter is then welded to the inner and outer bath tubes. This last proceedure is necessary to insure that the heating from the helium-arc welding does not damage the solenoid. Thus, the solenoid is completely sealed in the stainless steel bath by helium-arc welding. Although this seems like a very permanent arrangement, the face plate can still be removed if necessary by machining away the welds. Since the superconducting solenoid underwent an extensive testing program prior to its mounting in the closed bath, this technique was considered quite safe. In addition, the resulting assembly — once leak tight — would prove to be trouble free.

To facilitate the description of the assembling procedure, imagine that the RT (room temperature), 77, 4.2 and 0.9 K right angle radiation

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[7] Although one could use extremely thin windows with this geometry, no effort was made to do so since the experimental conditions did not warrant it.

shields are all removed at their vertical decoupling joint (in the region above the solenoid bath), and that the only item completely assembled is the ^{3}He tail (without the holmium sample). The solenoid bath assembly is then put in place and supported by the "solenoid support rod." At this point the stainless steel sleeve B is pushed up out of the way and the cup joint A is soldered. During this assembly a jig is used to accurately position the solenoid bath, so that when the holmium sample is in place, it is located in the center of the solenoid. A bakelite sleeve is used to hold the horizontal part of the 0.9 K radiation shield in place during the soldering of the cup joint A. The sleeve B is then moved down into position and secured. The solenoid bath feed tubes from the magnet bath are connected to the ones above using solder sleeve joints. This is done, of course, after the current and persistent switch leads as well as the teflon extension for S_1 are hooked up. The teflon extension for S_1 actually terminates in a 0.125 in. o.d. by 0.010 in. wall stainless steel tube which is welded to the inside bottom of the solenoid bath. All three solenoid bath feed tubes are off-centered so as not to be in the way of the neutron beam. The sample is put in next and the 0.010 in. window for the front of the 0.9 K radiation shield is soldered in at C and the vacuum space V_2 is checked for leaks. A photograph of the lower part of the cryostat at this point in the assembly is shown in figure 3.

Since the 4.2 and 77 K baths of the cryostat share a common vacuum, their radiation shields and windows can all be fastened by mechanical means rather than solder joints. Thus, the 0.010 in. thick front window for the solenoid bath, which is put on next, is done so using screws. The 77 K right angle radiation shield is made in two pieces with the front piece being attached about midway down the solenoid bath, see figure 2. With the back of the 77 K radiation shield just hanging on the solenoid bath, the right angle RT tail (with its large front window off) is put over it and brought to a few inches of its final position. The 77 K radiation shield is then manipulated into place and made fast. Then the RT tail is fastened. The front of the 77 K radiation shield (along with its 0.010 in. thick window) and the large RT front window

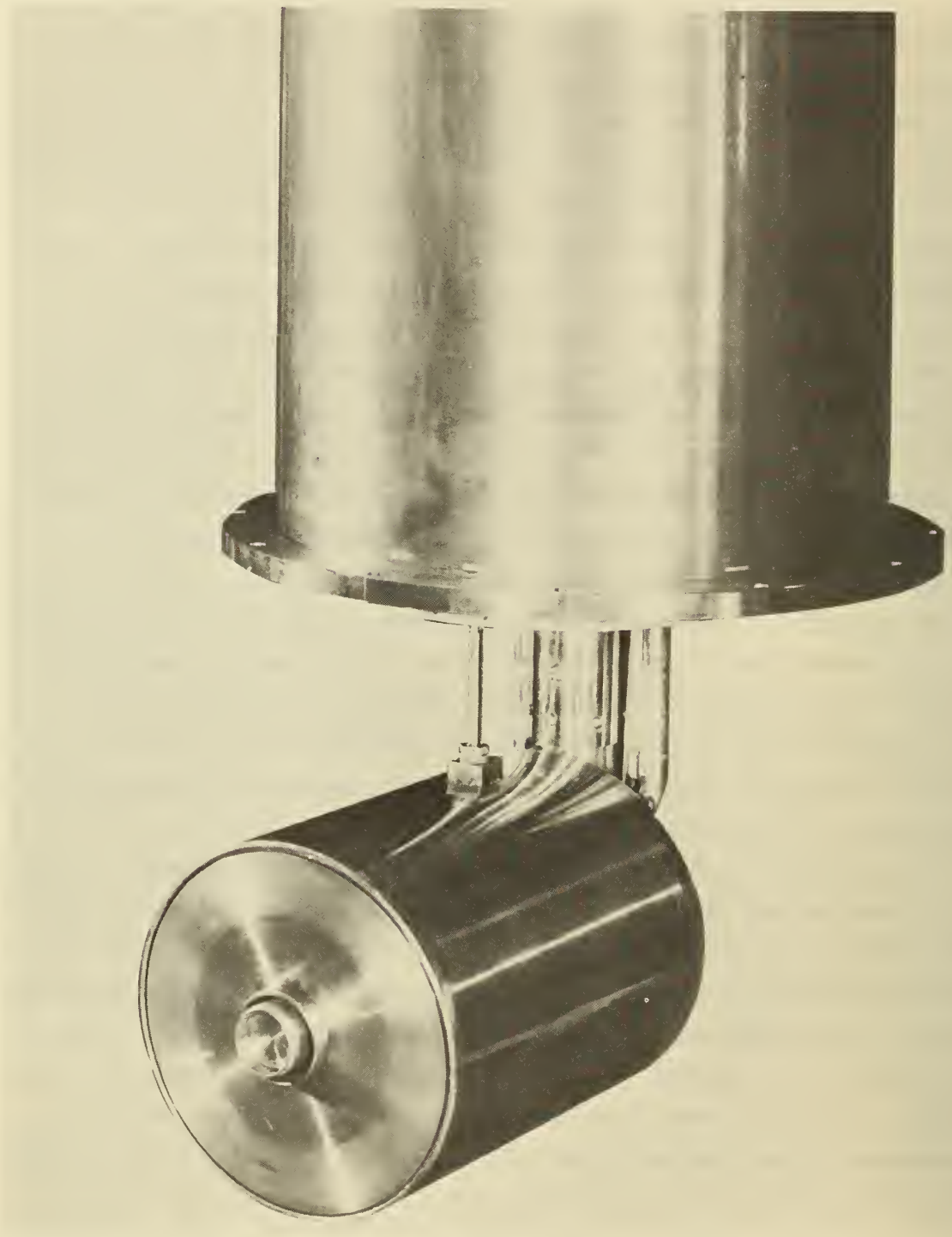

Fig. 3. Photograph of the lower part of the ^{3}He cryostat showing the superconducting solenoid bath and 0.9 K radiation shield.

are put on. Finally, the two small outside 0.030 in. stainless steel windows are put on completing the assembly.[8/]

The superconducting solenoid has an i.d. of 1.75 in., an o.d. of 5.0 in. and is 5.0 in. long. It could be operated as high as 4.38 MA/m (center field) in the persistent mode without going normal. At this field the stored energy is 5.1 kJ. The power and persistent switch leads from the solenoid terminate in a connector located in the bottom of the main 4.2 K bath. Removable leads are used from this connector to the top of the cryostat in order to reduce the heat leak into the 4.2 K bath when the solenoid is operated in the persistent mode. A 1 Ω - 10 W resistor which shunts the power leads is located near the connector in the main 4.2 K bath to help dissipate some of the energy away from the small solenoid bath if the solenoid should accidently go normal.

A photograph of the entire refrigerator with the cryostat fully assembled is shown in figure 4.

3. OPERATION OF THE ^{3}He REFRIGERATOR

After the cryostat is assembled the vacuum spaces V_1 and V_2 are pumped. During this time heater tapes are wrapped around the outside walls of the cryostat to speed-up the out-gassing. A heater is also used to out-gas the charcoal located on the 0.9 K bath. It normally

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8/ It is relatively easy to change the sample after the cryostat is assembled as it only involves removing the large RT window, the front part of the 77 K radiation shield, the 4.2 K window, and the 0.9 K soldered window. Lining up the sample with the neutron beam is also fairly straightforward for this type of cryostat as we can use standard visual techniques. The procedure is to remove all the inside windows in front of the sample and replace the outside window with a transparent one — thus the sample can be viewed directly. The cryostat can then be cooled to 77 K, at which temperature 97% of the vertical shrinkage (0.180 in.) has taken place, and then the sample can be lined up with the neutron collimator.

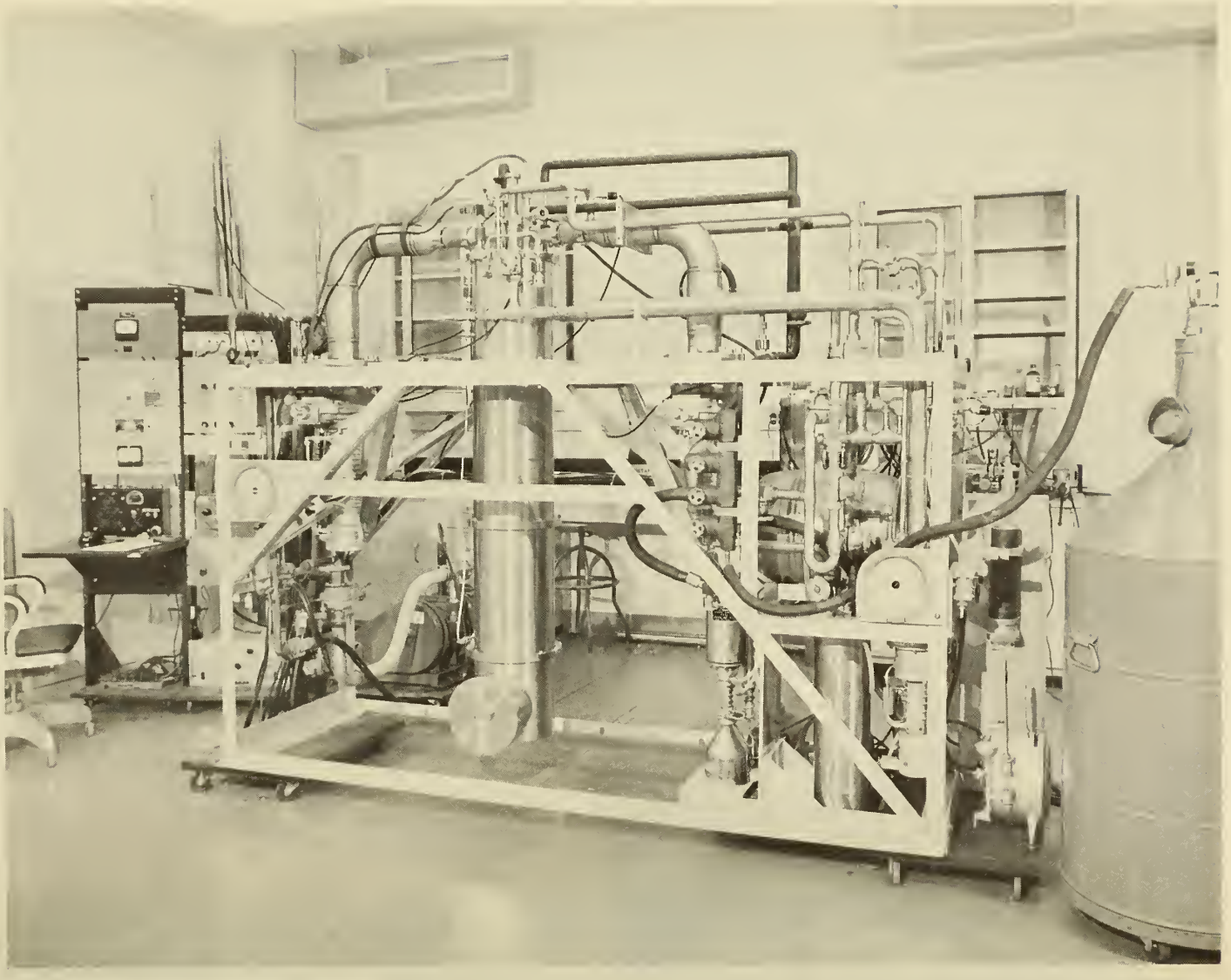

Fig. 4. Photograph of the complete ^{3}He refrigerator.

takes about 24 hours to reduce the pressure low enough ($\sim 10^{-5}$ mm of Hg) to start the precooling. However, before this is done ^{3}He exchange gas is vented into the vacuum space V_2. The cryostat is then precooled to 77 K by filling both liquid nitrogen baths, the large liquid helium bath, and the small pumped liquid helium bath with liquid nitrogen. During this time the small syphon S_2 is removed. Once the sample and the system are at 77 K (this is determined by the two carbon resistors mentioned previously, plus a similar carbon resistor mounted on top of the 0.9 K bath and a copper wire resistance thermometer wrapped around the 0.9 K bath)[9], the liquid nitrogen is removed by pressurizing the 4.2 K bath and having it retransferred out of the large syphon S_1. The liquid nitrogen is removed in a similar manner from the pumped ^{4}He bath by inserting a long tube to the very bottom. After this is done the small syphon is replaced. The entire precooling to 77 K takes about one hour and consumes approximately 80 liters of liquid nitrogen. Liquid helium is then transferred into the 4.2 K bath. This transfer is done rather slowly at first ($\sim$ ½ liter/minute) so that maximum use of the cold helium gas is obtained. Once the solenoid is superconducting, the transfer rate is increased to about 1 liter/minute. When there is sufficient liquid in the main helium bath ($\sim$ 5 - 6 inches above the bottom plate) the small syphon is opened and the 0.9 K bath is partially pumped. In this way the sample, ^{3}He tail and 0.9 K bath are cooled from 77 to 4.2 K. The pumped ^{4}He bath is then filled. During all this time the main helium transfer is still going on. When the 4.2 K bath has approximately 18 liters in it, the transfer is stopped. The time required for the complete helium transfer is also about one hour and about 50 liters are used.

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[9] The copper resistor is wound non-inductively from No. 40 wire and such that its total resistance is about 500 Ω at room temperature. Its resistance versus temperature relation is almost linear down to about 50 K and then begins to "flatten out." However, it is still a useful thermometer to 4.2 K.

The next step in the operation of the refrigerator depends upon what type of data is to be taken with the holmium sample; that is, either unpolarized or polarized data. Since holmium is ferromagnetic, once it is magnetized hysteresis effects will show up as a small but measurable polarization at 0.3 K, even in zero magnetic field.[10/] Thus, all the zero polarization data must be taken before the magnet is energized.[11/] Assuming this has been done the magnet is next energized to its rated field and put in the persistent mode. At this point the main power leads can be removed to reduce the heat leak into the 4.2 K bath. The small ^{4}He bath is pumped on next. While the temperature of this bath is being reduced from 4.2 to 0.9 K, the ^{3}He system is activated. ^{3}He gas from the storage tank is vented into both sides of the frit; that is, the ^{3}He pump line and the ^{3}He return line (normal condensing side). This technique of condensing the gas on both sides of the frit reduces the start-up time for circulating ^{3}He. When the temperature of the small ^{4}He bath reaches approximately 1.0 K, the ^{3}He exchange gas in V_2 is pumped out. After about 10 minutes the ^{3}He mercury pump is started and the ^{3}He cooling cycle begins. The ^{3}He gas from the exhaust side of the mercury pump is returned through a nitrogen-cooled trap to the condensing line (the storage tank being isolated from the system). When the temperature of the ^{3}He tail reaches about 0.4 K, the ^{3}He oil diffusion pump is used with the mercury pump backing it in order to obtain a

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10/ Holmium metal is paramagnetic down to 130 K (Néel temperature) where it becomes antiferromagnetic with a helical spin structure. At 20 K it becomes ferromagnetic along the c-axis — still maintaining the antiferromagnetic spin structure in the basal plane. Thus, if the sample is accidently magnetized it has to be then warmed up above 133 K in order to be sure it is completely unmagnetized when cooling down.

11/ Data have to be taken at temperatures of 4.2 and 0.32 K for zero nuclear polarization in order to make certain that there are no "nuclear effects" due to just cooling the sample.

a final temperature of 0.32 K.

If the refrigerator is operated in a "one-shot mode", namely, returning the ^{3}He gas from the mercury pump to the storage tank rather than the condensing line, the temperature can be reduced by 20 to 30 mK. However, the disadvantage of this method of operation is that after 8 - 12 hours all the ^{3}He gas is pumped back into the storage tank and the sample warms up.

The temperature of the sample is determined by the two carbon resistors mentioned previously; one in the front face of the holmium sample and the other in the copper part of the ^{3}He tail. Carbon resistors were chosen over germanium because they exhibit a much smaller magnetoresistive effect. The disadvantage of carbon resistors is that their calibration can change slightly when cycling between room temperature and liquid helium temperatures. Their temperature dependence is given by the following logarithmic equation:

$$T = \frac{A \log R}{(\log R - B)^2} ,$$

where the constants A and B are determined from two accurately known temperatures (usually 4.2 and 0.9 K). The generated curves for the resistor mounted in the ^{3}He tail are shown in figure 5 for the case of zero magnetic field and also for H = 3.58 MA/m.[12] Its temperature dependence below 0.9 K is checked against the vapor pressure of ^{3}He by assuming it is in good thermal contact with the liquid.[13], [14] This assumption has been verified in previous work with similar resistors —

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[12] This is the field at the center of the solenoid, that at the resistor is ~ 0.5 MA/m.

[13] As pointed out previously the surface area of the bottom of the inside of the ^{3}He tail has been increased substantially.

[14] The thermomolecular pressure correction has been taken into account using the data of T. R. Roberts and S. G. Sydoriak [3], in determining our low temperature points.

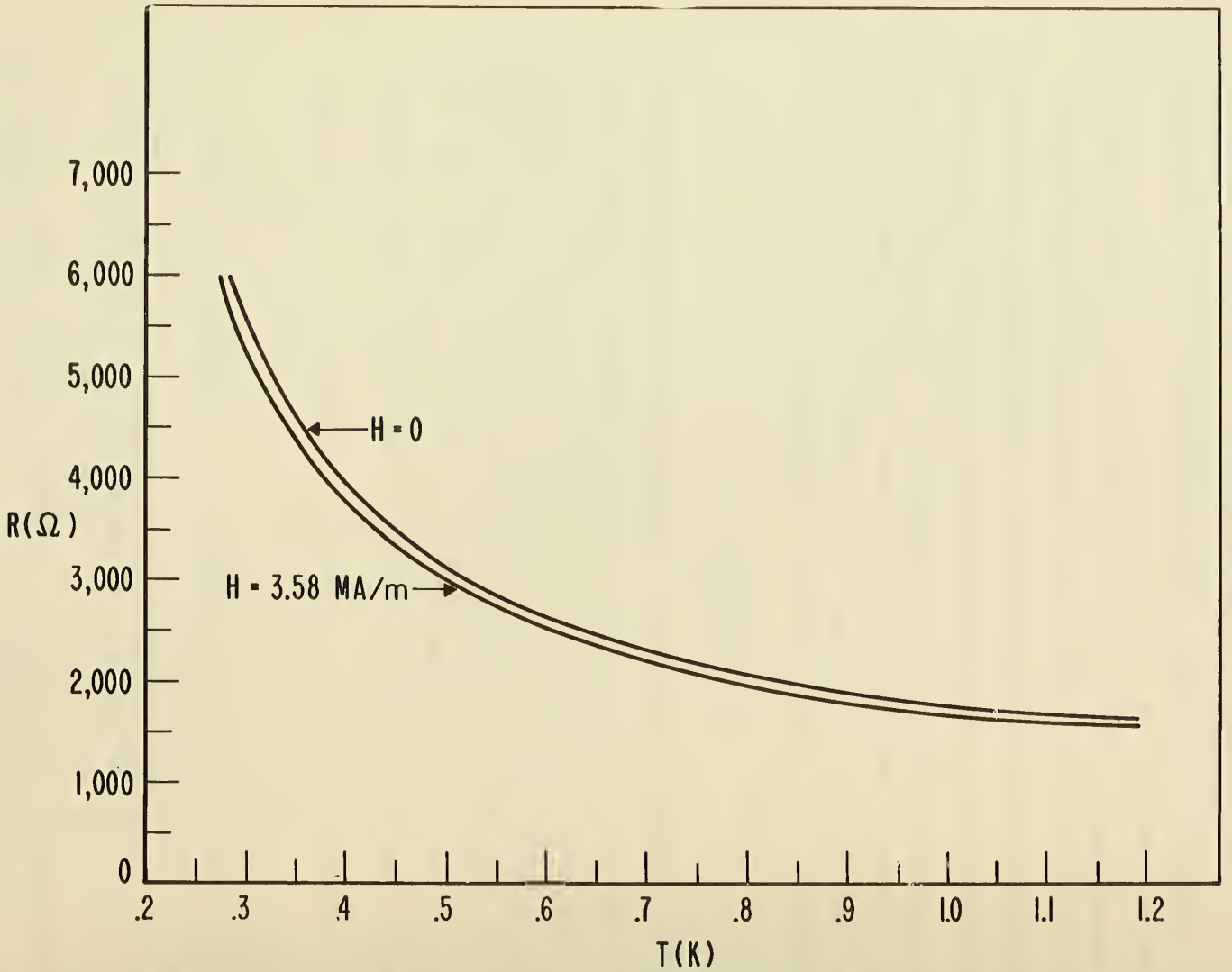

Fig. 5. Resistance versus temperature curves for a 470 Ω Speer carbon resistor (grade 1002). The field given (3.58 MA/m) is for the center of the solenoid, that at the resistor is about 0.5 MA/m.

one mounted directly in the ^{3}He liquid. Although the resistor mounted in the front face of the sample has additional thermal boundary resistances (the greased threaded joint and the holmium to copper solder joint) between it and the resistor in the ^{3}He tail, their temperatures agree to within 5 mK when the final temperature is reached. The uncertainty in the final temperature of the sample is estimated to be ± 10 mK.

The cooling capacity of the ^{3}He cycle is determined solely by the pumping characteristics of the system; that is, just the through-put of the ^{3}He gas. Although this can be calculated, the error in such calculations may be as large as 50%. Measurements can be made by replacing the sample with a heater. This has been done for a pumping system similar to ours and the results were that the cooling capacity is approximately 2 mW down to 0.4 K and then falls off to zero at 0.26 K; see figure 3 of reference [2]. The time it takes to cool our holmium sample (2.5 moles) during the ^{3}He cycle depends upon its heat capacity[15/], its thermal contact to the ^{3}He, the heat leak into it, and the cooling capacity of the refrigerator. If we just consider its heat capacity and use the cooling curve of reference [2], calculations show that it should take about 1.5 hours to cool it from 1.0 to 0.32 K. This value agrees fairly well with what we measure (1.5 to 2 hours) — indicating that we are making good thermal contact with the sample and that the heat leak into it is rather small.

The total time needed from the beginning of the ^{4}He transfer to when the sample is at 0.32 K is 3 - 4 hours. Once the sample is at 0.32 K the refrigerator can maintain this temperature for 2 - 3 days without retransferring any liquid helium. The only servicing required is keeping the liquid nitrogen traps full — this is usually accomplished by an automatic filling system. Retransferring into the large 4.2 K bath is done before the liquid level drops below the bottom plate,

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15/ The extremely large hyperfine field (~ 700 MA/m) in holmium metal gives rise to a Schottky type specific heat anomaly which has a maximum of 7 J/mole at ~ 0.3 K.

thus assuring us that the superconducting solenoid does not go normal. This retransfer usually goes quite fast and uses a minimum of liquid as the magnet and the bath are at 4.2 K. If data are to be taken at 4.2 K the sample can be warmed up to this temperature in 20 minutes. The procedure is to vent ^{3}He exchange gas into the vacuum space V_2 and warm up the pumped ^{4}He bath. The latter is accomplished by simply closing the valve to the pump and retransferring 4.2 K liquid from the large bath using the small syphon S_2.

If the sample has to be changed, the entire system can be warmed up in a few hours by venting dry nitrogen gas into both vacuum spaces V_1 and V_2. The screws holding the outside windows are loosened so that when V_1 reaches 1 atmosphere the windows "pop out." Nitrogen gas is continuously blown out at a very slow rate to insure that no moisture gets into the cryostat. Once the cryostat reaches room temperature the sample can be changed and the "start up" procedure described previously is begun. The total time for changing samples and getting "cold" again takes about 36 hours.

4. CONCLUSIONS

After the ^{3}He refrigerator with the new cryostat was tested the entire system was dismantled and shipped by air freight to the Atomic Energy Research Establishment, Harwell, England. The first experiment was a measurement of the effect of nuclear deformation on the total neutron cross section of ^{165}Ho over the energy range of 2 to 135 MeV.[4] This was scheduled at the Harwell synchrocyclotron as it is the only existing source of neutrons covering such an extensive range in energy. The experiment was to commence 11 days after the equipment arrived in England — it was completely assembled and operational 2 days ahead of schedule. The measurements took 10 days of machine time. Additional data measuring the effect as a function of temperature were taken 3 weeks later. The entire experiment took less than 2 months. In contrast previous work done at only four discrete neutron energies using Van de Graaff accelerators took from 1 to 2 years. [5-8]. Thus, this experiment demonstrated the feasibility of using such transportable

oriented nuclear targets at other laboratories.

The cryostat alone was shipped back to the United States where it was modified to accept a larger bore superconducting solenoid to accommodate a larger diameter holmium sample. This was used for a second experiment at Harwell, this time at their linear accelerator.

The right angle geometry of the bottom part of the cryostat can easily be included in the design of a ^{3}He - ^{4}He dilution refrigerator. With such a refrigerator, incorporating a very high field superconducting solenoid, one could obtain quite large polarizations for many nuclei using the "Brute Force" technique.

It is a pleasure to acknowledge helpful discussions with Mr. R. S. Kaeser and members of the Cryogenic Physics Section. We would like to thank the N. B. S. Instrument Shops for their help in fabricating various components of the cryostat, and in particular, thank Mr. R. R. Orwick for his very fine helium-arc welding. The assistance of Mr. D. F. Martin in the later modification is deeply appreciated.

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